Nanotechnology **9** (1998) 331–336. Printed in the UK PII: S0957-4484(98)90319-1

V-shaped metallic-wire cantilevers for combined atomic force microscopy and Fowler–Nordheim imaging

Charles A Peterson†, Richard K Workman, Xiaowei Yao, Jeffery P Hunt and Dror Sarid

Optical Sciences Center, University of Arizona, Tucson, AZ 85721, USA

Received 6 January 1998, in final form 14 May 1998

Abstract. A method for fabricating V-shaped cantilevers from a flattened Pt/Ir metal wire for combined atomic force microscopy and Fowler–Nordheim imaging is described. These novel cantilevers have been found to be more robust then conventional ones used for scanning capacitance and magnetic force microscopy as their conductivity is maintained even after a large number of surface scans. The use of a V-shaped geometry improves on earlier single-beam geometries by reducing rms imaging noise. Characterization of these cantilevers and combined atomic force microscopy and Fowler–Nordheim images are reported.

1. Introduction

A novel mode of operation of a conducting-tip atomic force microscope (CT-AFM), based on Fowler–Nordheim (FN) field-emission, has been shown to be a useful tool for characterizing thin dielectric films fabricated on semiconductor surfaces. In particular, it has been demonstrated that one can map local variations in silicon oxide thickness and dielectric strength across a silicon substrate [1–4]. An important factor in making this technique a viable commercial tool is that tips withstand the intense electric and mechanical stresses required, even after a large number of line scans, and so maintain their sharpness and conductivity. These stresses originate from operating in the FN field-emission regime where detectable currents require that fields greater than 10 MV cm^{-1} be applied between tip and conducting substrate. Fields of such strength are capable of evaporating metallic coatings commonly used on tips intended for capacitance and magnetic measurements as well as creating forces strong enough to cause tip breakage.

Commercially available conducting tip cantilevers are fabricated by coating either silicon or silicon nitride cantilevers with thin metal films or by doping silicon cantilevers. Using coated tips necessitates methods with slow scan rates across hard surfaces such as SiO_2 to minimize wear, and spurious voltage or current spikes may lead to their catastrophic failure. Highly doped cantilevers with a high aspect ratio, although yielding superior AFM images, induce highly non-uniform electric fields which is undesirable for FN imaging, are prone to rapid oxidation, and are expensive. An attractive alternative to these cantilevers is made possible by using solid-metal cantilevers for which wear, evaporation, oxidation and restructuring have a small effect on shape and conductivity. However, single-beam solid metal cantilevers, as depicted in figure 1(*a*), suffer from rotation about the x-axis that gives rise to noisy and distorted images. On the other hand, using V-shaped solid-metal cantilevers, fabricated from a flattened Pt/Ir wire, figure 1(*b*), alleviates this problem. In this paper we report on the mechanical and electrical characterization of such cantilevers and on their use for combined AFM and FN imaging.

† Email address: capeters@physics.arizona.edu

2. Tip fabrication

To fabricate a V-shaped cantilever, one starts with a 80%/20% platinum-iridium wire, commonly used in scanning tunnel microscopy (STM), that has been flattened to 100 μm$\times$50 μm by the supplier. Further flattening of the wire is accomplished by pressing it between a stainless-steel rod and plate. The flattening is necessary in order to lower the spring constant and to produce a thin, wide working surface that can be cut easily. Using a magnifier and a sharp razor blade, the wire is split length-wise for a distance of 2 mm and the two beams separated to form a V-shaped structure. Further flattening helps to smooth out burrs and make the beams more uniform, while careful trimming improves the uniformity of the cross section. Note that the single-beam cantilevers fabricated for this study are produced by the same method with the exception of the beam cutting step. These beams are then soldered to a small piece of copper about the size of a conventional cantilever substrate, to provide a stable mount for the cantilever as well as an excellent electrical contact. The cantilever is

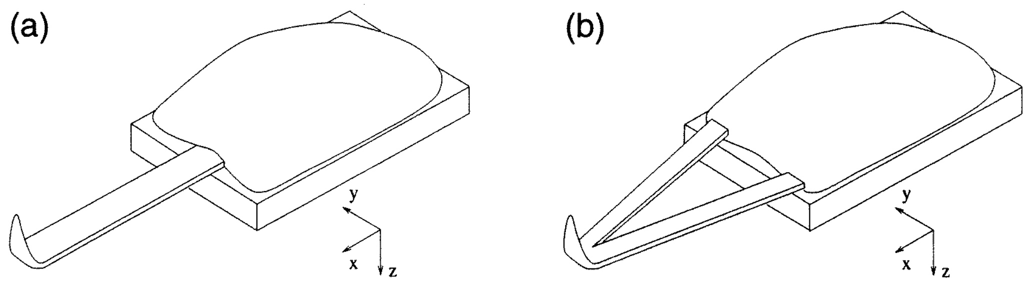

Figure 1. Line art diagram of (*a*) a single-beam metallic-wire cantilever and (*b*) a V-shaped metallic-wire cantilever and definition of axes.

then clipped from the remaining wire about 1 mm from the point of the 'V'. The cantilever substrate is next clamped to a micrometer and lowered into a solution of 2M $CaCl_2$. First, the top side ($+z$-direction) of the cantilever is etched by bringing the wire into contact with the solution and then retracting it slightly, using surface tension to limit the reaction location to the top, and then applying 10 V AC for about 1–2 min. This step both thins and narrows the wire with a subsequent reduction of the spring constant. It also rounds and polishes the back of the cantilever which not only provides a good reflecting surface but also spreads the AFM laser beam into a line, reducing the effect of cantilever rotations. To form the tip, the cantilever is placed on the edge of an aluminum block and the overhang bent with the back of a razor blade. The apex of the tip is formed by placing it in the $CaCl_2$ solution and etching it at 30 V AC until the reaction ceases.

Figure 2 shows four scanning electron micrographs (SEM) of a completed V-shaped cantilever. Figure 2(*a*) is a top view of the cantilever and a portion of the copper substrate, where the tip apex is on the right, pointing out of the page. Figure 2(*b*) is a side view of the cantilever and substrate with the apex of the tip on the left, pointing up. Figure 2(*c*) is a close-up image of the tip's apex taken from the side before FN imaging, while figure 2(*d*) is a similar view of the cantilever taken from the front after FN imaging.

3. AFM characterization

Several parameters have to be considered in the characterization of a cantilever: the spring constant, k, that must be small enough so as not to modify the sample during a scan, the resonance frequency, f_0, that must be high enough to enable reasonably fast scan rates, and the tip radius of curvature, R, that should be small to minimize convolution effects. This last property is particularly important when scanning tall or deep features and less so when scanning nearly flat surfaces. However, during FN imaging, an extremely small R produces intense, non-uniform electric fields at the tip apex that may damage the tip and sample. A larger R, on the other hand, offers a larger effective tunneling area and produces a more uniform field. Closely related to R is the tip cone angle, θ, which is the angle of the tip shank as it leaves the tip apex and approaches the cantilever. A sharp angle is important when reaching into surface depressions and in maintaining a reasonable curvature as the tip wears off. Finally, in order to obtain clear AFM images, the rms imaging noise must be low.

The spring constants of the fabricated V-shaped cantilevers were obtained by comparing force curves on a hard surface and on a surface with a known spring constant. The hard surface was used to calibrate cantilever deflection during the force curves. A 9 mm long, Pt/Ir wire with a 100 μm $\times$ 50 μm cross section was then attached to a sample puck and the tip was engaged onto the end of this wire. Using a *Mathematica* model [5] it was found that such a wire has a spring constant of $k = 1$ N m^{-1}. From a force curve performed on this wire the spring constants for two cantilevers were found to be $k = (9 \pm 2)$ N m^{-1} and $k = (3 \pm 0.5)$ N m^{-1}. Using a similar technique on a single-beam cantilever fabricated from the same wire we found $k = (2.3 \pm 0.5)$ N m^{-1}.

A second *Mathematica* model [5], using the geometries of figure 2, yielded resonance frequencies of 4.1 and 2.1 kHz for the two V-shaped cantilevers. In order to measure the actual resonance frequency, the cantilever was engaged onto a sample of highly-ordered pyrolytic graphite (HOPG) and then withdrawn 10 μm. An AC voltage of 0.5 V was applied between the tip and sample, causing the cantilever to oscillate, and the signal from the AFM photodetector was routed to a spectrum analyzer. The resonance frequencies for the two V-shaped cantilevers were found to be 4.95 ± 0.01 and 2.10 ± 0.01, and 3.85 ± 0.01 kHz for the single-beam cantilever.

The radius of curvature of the tip in both the x- and y-directions was determined by scanning a grating at 0° and 90°. Assuming that the tip is a sphere and the edge of the grating is perfectly vertical, one can estimate the radius of curvature using $R = (h^2 + v^2)/2v$. Here h is the horizontal distance from the edge of the grating where the tip begins to fall off to the point where it settles to the bottom of the grating, and v is the vertical distance with the same criteria. With this method, the radius of curvature of one tip was determined to be 1 μm in the x-direction and 3 μm in the y-direction, while the other tip had radii of 3 and 10 μm, respectively. The cone angles in the x- and y-directions, using SEM images, yielded $\theta = 20°$ and 35° respectively for one tip, and $\theta_{x,1} = 45°$ and 45° respectively for the other.

The primary advantage of using a V-shaped cantilever is the increased torsional spring constant compared with a

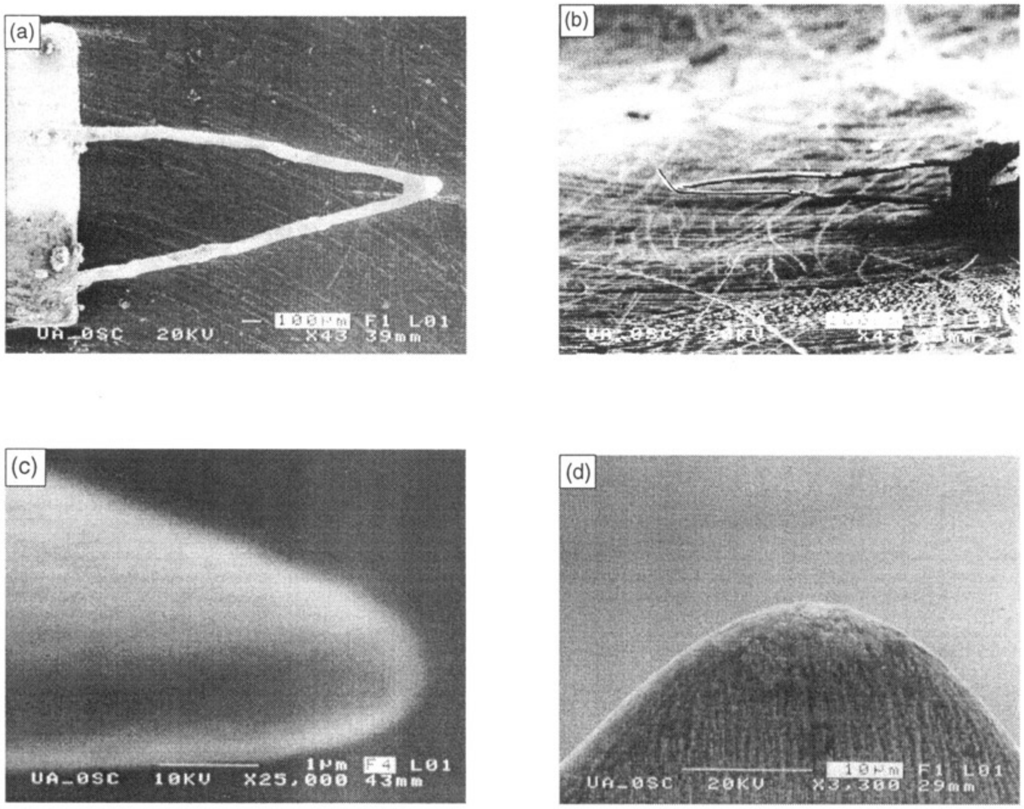

Figure 2. SEM of a metallic V-shaped cantilever. (*a*) Top view of cantilever (tip on right-hand side, pointing out of the page) and substrate. (*b*) Side view of cantilever (tip on left-hand side, pointing up) and substrate. (*c*) Close-up of tip apex from the side. (*d*) Close-up of tip apex from the front.

single-beam cantilever. A low spring constant for rotations about the x-axis allows the tip apex to travel along the sample surface perpendicular to the scan direction, causing loss of definition of surface features. Rotations also direct the AFM laser beam away from the detector, increasing rms imaging noise. As a test of the quality of AFM images produced by V-shaped metallic-wire cantilevers, we used one to scan a grating with a step height of 9.3 nm and a pitch of 0.91 μm. Figure 3(a) is an AFM image of the grating produced by the new cantilever. For comparison, figure 3(b) is an image of the grating produced with a single-beam cantilever. Clearly the single-beam cantilever image lacks definition while the edges of the V-shaped cantilever image are sharp. To measure the rms imaging noise we scanned on samples of HOPG, and for the two V-shaped cantilevers found values of 2.8 and 2.5 Å as compared with 10 Å for the single-beam cantilever.

4. Electrical characterization

Several methods were employed to study the electrical characteristics of the metallic-wire cantilever. To test the electrical contact at the apex, the tip was placed in contact with a HOPG sample and I–V curves were performed. In most cases the measured curves were highly linear over the –500–500 pA range with a slope corresponding to a 10 GΩ resistor which was placed in series with the sample. Occasionally, curves displayed slight tunneling behavior, i.e. an exponential rise in current which approaches the previously mentioned linear region. Such curves were likely to be due to the formation of thin insulating layers on the apex of the tip during scanning, but after a few ramps to ± 4 V, the layer was removed and the curves returned to essentially linear curves.

To determine the force required for obtaining a solid contact, a constant bias was applied between the tip and sample during force curve measurements to find the current as a function of force as shown in figure 4. One observes that as the sample approaches the tip (black), electric, van der Waals, and other forces cause the tip to snap into contact. The closing of the tip–sample contact is initially accompanied by a current spike which is likely due to the charging of parasitic capacitances which decay to a constant current corresponding to the 10 GΩ resistor. The formation of a conducting path between the tip and sample clearly shorts out the electric field. As the sample retracts (grey), the remaining forces maintain the tip–sample contact until a critical negative force is reached. Just before the tip snaps off the sample, there is a decrease in the observed current as the contact area is reduced. Finally, the tip snaps off, the current returns to zero, and the electric field is re-established. Therefore, with metallic tips, good electrical contact is easily maintained.

The FN tunneling current is given by [6]

$$I_{\mathrm{FN}} = A_{\mathrm{eff}} \frac{e^3}{8\pi h\phi} F^2 \exp\left(\frac{-8\pi\sqrt{2m_{\mathrm{eff}}}}{3he}\frac{\phi^{3/2}}{F}\right). \qquad (1)$$

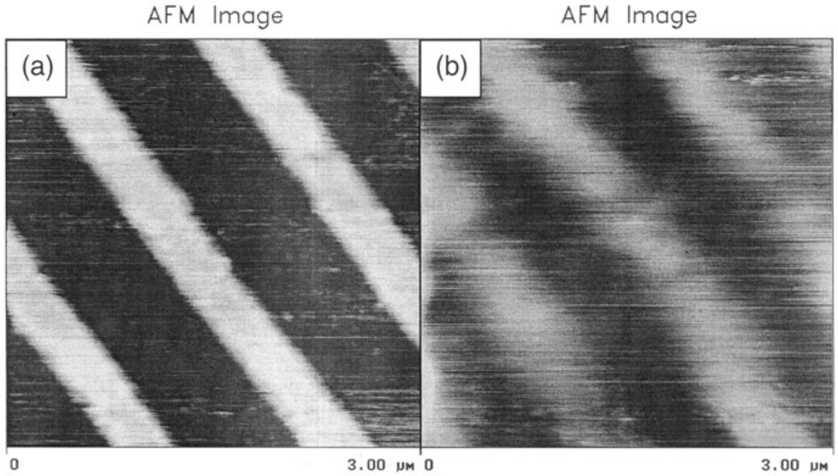

Figure 3. Comparison between V-shaped and single-beam cantilevers. (*a*) AFM image taken at a 45° scan angle on a grating (step height: 9.3 nm, pitch: 0.91μm) with a V-shaped metallic-wire cantilever. (*b*) AFM image of grating taken with a single-beam metallic-wire cantilever.

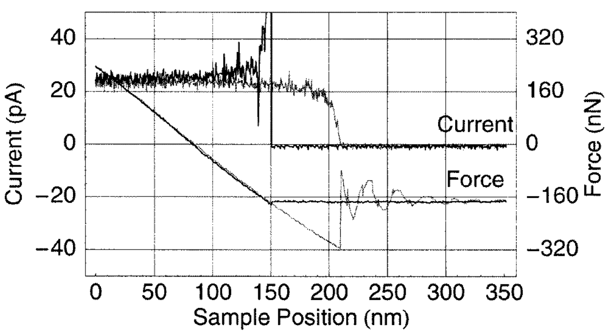

Figure 4. Current (top) and force (bottom) as a function of sample translation, where the sample approach is plotted in black and the return is plotted in grey. Note that solid-metal cantilevers provide excellent electrical contact even at negative forces.

Here, F is the applied field; ϕ the metal-oxide barrier height; A_{eff} the effective electrical contact area; and m_{eff} the effective electron mass. Values for ϕ and m_{eff} are determined by the material of the tip and sample and a reasonable $\sqrt{m_{\mathrm{eff}}}\phi^{3/2}$ product can be found for a particular material pair by fitting the FN tunneling equation to I–V curves taken on large-scale MOS capacitors of a known area. On the other hand, the tunneling current is only weakly dependent on A_{eff}, therefore fitting produces unreliable values for the electrical contact area. The physical contact area may be determined using the Hertz elastic contact theory [2], however, this area may be enhanced by residual water vapor forming a meniscus at the tip–sample contact. Instead, one observes that on thin oxides, where lateral tunneling is less likely, A_{eff} is closely related to the horizontal FN resolution. To measure the FN resolution, the CT-AFM system was used to locally stress the oxide by applying a constant 2 pA current while scanning a 100 nm $\times$ 100 nm uniform 4 nm oxide. To prevent sample oxidation, the experiment was performed in dry nitrogen. Figure 5(*a*) consists of a 1 μm $\times$ 1 μm AFM image (left) of the oxide after stressing, showing that the oxide remains flat, and a simultaneous, constant 0.5 pA current FN image of the oxide. Figure 5(*b*) shows a cross section of the stressed location from figure 5(*a*), where the smallest feature demonstrates a resolution of approximately 20 nm suggesting that $A_{\mathrm{eff}} \approx 300$ nm^2. Figure 5 also demonstrates the ability of the V-shaped metallic-wire cantilevers to obtain FN images without loss of tip conductivity due to wear, evaporation, oxidation and restructuring while simultaneously capturing the AFM image [7].

5. Discussion

Table 1 summarizes the properties of the two V-shaped metallic-wire cantilevers examined in this study as well as the properties of single-beam metallic-wire cantilevers and ranges of values for commerical cantilevers. Although the AFM imaging quality of the handmade metallic-wire cantilevers is inferior to commercial coated silicon and silicon-nitride ones, they are expected to perform more reliably at high electric fields, maintain better electrical contact with the sample after repeated scans, and should recover from a catastrophic tip failure. Comparing the properties of the single-beam and V-shaped metallic-wire cantilevers indicates that values for k, f_0, R, θ and A_{eff} are similar owing to the similarity in the method of their production. For the second V-shaped cantilever, the experimental value of f_0 agreed with the value obtained from the model to within the estimated experimental error. The first cantilever did not, however, probably due to an underestimation of the non-uniformity of the cantilever dimensions. Some control over k and f_0 is possible by varying cantilever length and back-side etch time. The values of R, θ and A_{eff} are determined by the apex etching process and tip wear, and are more difficult to control. Though not examined in this work, careful trimming of the tip shank with a razor blade before etching may improve these values. The advantage of using a V-shaped cantilever over a single-beam cantilever is the

Table 1. Summary of AFM and FN properties of two V-shaped metallic-wire cantilevers and comparison with properties of single-beam and commercial cantilevers.

	V-Shaped no 1	V-Shaped no 2	Single-beam	Commercial
k_{exp} (N m^{-1})	9 ± 2	3 ± 0.5	2.3 ± 0.5	5–50
$f_{0,theory}$ (kHz)	4	2.1	N/A	N/A
$f_{0,exp}$ (kHz)	4.95 ± 0.01	2.10 ± 0.01	3.85 ± 0.01	5–50 kHz
R_{tip} (μm)	1, 3	3, 10	Similar	5–50 nm
θ_{tip}	20°, 35°	45°, 45°	Similar	20°
RMS noise (Å)	2.8	2.5	10	≈ 0.5
A_{eff} (nm^2)	N/A	1000	Similar	N/A

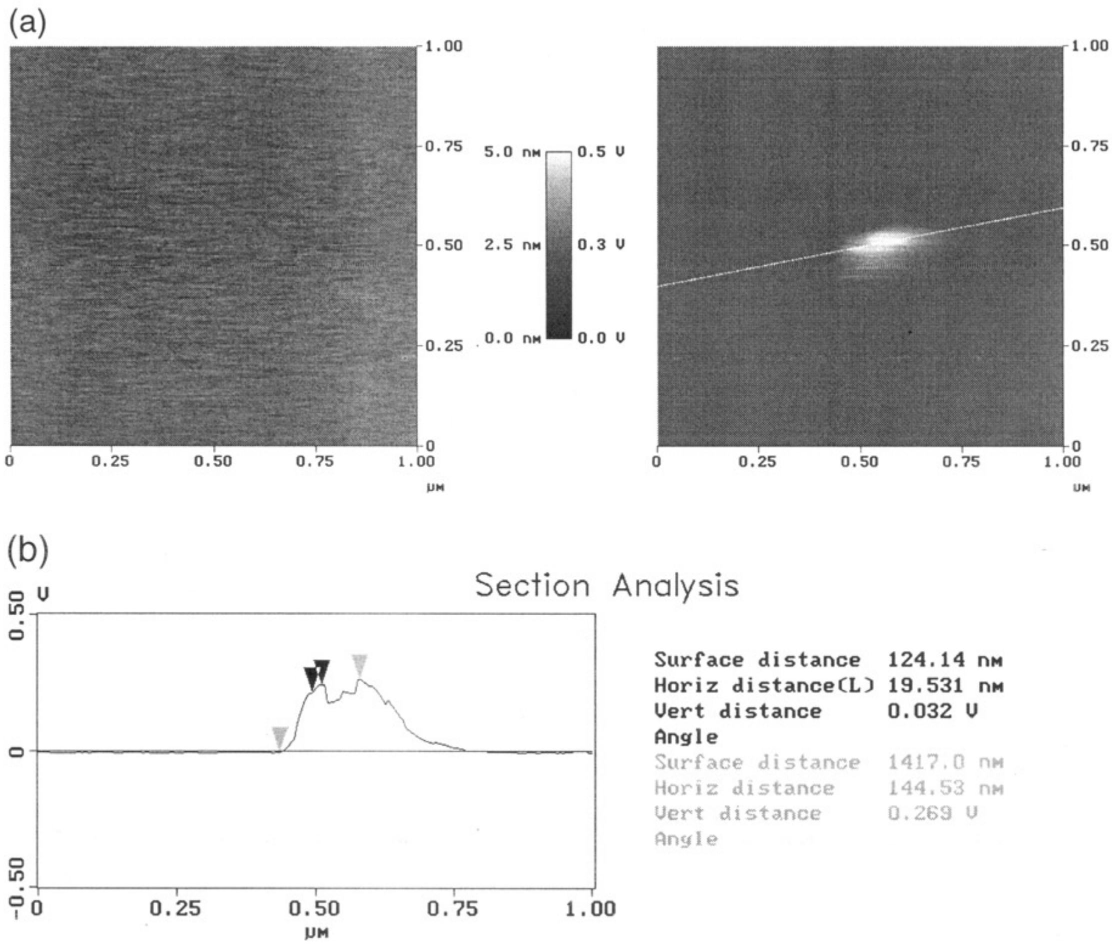

Figure 5. (*a*) Simultaneous AFM (left) and constant-current FN (right) images of an oxide locally stressed with FN emission. The AFM image confirms that no oxide was grown during the stressing, maintaining a flat sample surface. Approximately 25 V (offset not shown on scale) was required to maintain a constant 0.5 pA current during the scan, while the stressed location required a 0.27 V increase. (*b*) A cross section of the FN image through the stress feature demonstrates a FN resolution of 20 nm.

improvement in AFM image quality as seen in figure 3, and the reduction in rms imaging noise as can be seen in table 1. Further improvements in signal-to-noise ratio may be gained by shortening the cantilever length. Halving this length, for example, doubles the cantilever deflection angle while scanning a feature, resulting in doubling the signal. However, halving the cantilever length increases k by a factor of eight unless a longer etching time is used to remove a corresponding amount of material.

6. Conclusion

A method for fabricating and testing V-shaped metallic-wire cantilevers has been described, and simultaneous AFM and FN images obtained. It was shown that using V-shaped, rather than single-beam, cantilevers substantially reduces imaging noise on AFM scans while maintaining all the advantages of solid-metal cantilevers desired for FN scans.

Acknowledgments

Support for this research has been provided by the Augmentation Award for Science and Engineering Research Training (AASERT), the Army Research Office (ARO), the Center for Microcontamination Control (CMC), and Digital Instruments. The authors would also like to thank Dennis Adderton for helpful discussions.

References

[1] O'Shea S J, Atta R M, Murrell M P and Welland M E 1995 *J. Vac. Sci. Technol.* B **13** 1945
[2] Ruskell T G, Workman R K, Chen D, Sarid D, Dahl S and Gilbert S 1996 *Appl. Phys. Lett.* **68** 93
[3] Ebersberger, Boit, Benzinger and Gunther 1996 *Int. Reliability Symp. (Dallas, TX)*
[4] O'Shea S J, Atta R M and Welland M E 1995 *Rev. Sci. Instrum.* **66** 2503
[5] Sarid D 1997 *Exploring Scanning Probe Microscopy using Mathematica* (New York: Wiley–Interscience)
[6] Fowler R H and Nordheim L 1928 *Proc. R. Soc.* A **119** 173
[7] Digital Instruments, Inc., Santa Barbara, CA, Nanoscope III AFM system